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Indian Standard
SPECIFICATION FOR CITRAL
(First Revision)

UDC 665·533·1

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *November* 1982

AMENDMENT NO. 1 JUNE 1992
TO
IS 1799 : 1981 SPECIFICATION FOR CITRAL

(First Revision)

(*Page* 3, *clause* 0.3) — Insert the following new clause after **0.3**:

'**0.3.1** Although in accordance with the general decision of the Committee, GLC method is preferred to wet method in case of aromatic chemicals, however, in case of citral both the methods, gas chromatography as well as bisulphite solubility are being retained because in citral, there is a possibility of presence of naturally occurring polymeric material or high boiling adulterants which are not resolved by GLC. On the other hand there is a possibility of addition of other aldehydes as adulterants in citral which may not be detected by wet method. Therefore, inclusion of both the methods in this standard is essential and complimentary to each other.'

[*Page* 6, *Table* 1, *Sl No.* (vi), *col* 2] — Substitute the following for the existing matter:

'Citral content by GLC, percent by mass, *Min.*'

(PCD 18)

Reprography Unit, BIS, New Delhi, INDIA

AMENDMENT NO. 2 JANUARY 1998
TO
IS 1799 : 1981 SPECIFICATION FOR CITRAL

(First Revision)

(*Page* 4, *clause* **0.8**) — Delete and renumber the subsequent clause.

(*Page* 4, *footnote marked* '*') — Delete.

(*Page* 5, *clause* **2.1**) — Substitute 'IS 6597 : 1988*' *for* 'IS : 6597 - 1972*'.

(*Page* 5, *clause* **4.2**, *line* 3) — Substitute 'IS 326 (Part 6) : 1986‡' *for* 'IS : 326 - 1968‡'

(*Page* 5, *clause* **5.1**, *line* 2) — Substitute 'IS 326 (Part 1) : 1984**' *for* 'IS 326 : 1968‡'.

(*Page* 5, *clause* **6.1**) — Substitute the following for the existing clause:

'**6.1** Test shall be conducted as prescribed under **4.1**, **4.2** and **4.3** and the appropriate references to relevant parts of these standards are given in col 4 of Table 1.'

(*Page* 5, *clause* **6.2**) — Substitute 'IS 1070 : 1992‖' *for* 'IS : 1070 - 1977‖'.

(*Page* 5, *footnote marked* '*') — Insert '(*first revision*)' at the end of the sentence.

(*Page* 5, *footnote marked* '‡') — Substitute the following for the existing title:

'‡Methods of sampling and test for natural and synthetic perfumery materials : Part 6 Determination of solubility (*second revision*) '

(*Page* 5, *footnote marked* '‖') — Substitute 'Reagent grade water (*third revision*)' for the existing.

(*Page* 5, *footnotes*) — Insert the following at the end:

'**Methods of sampling and test for natural and synthetic perfumery materials : Part 1 Sampling (*second revision*).'

[*Page* 6, *Table* 1, *Sl No.* (ii), *col* 5] — Substitute 'IS 2284 : 1988†' for the existing.

[*Page* 6, *Table* 1, *Sl No.* (iv), *col* 5] — Substitute 'IS 326 (Part 5) : 1986||' for the existing.

(*Page* 6, *Table* 1, *footnote marked* '†') — Insert '(*first revision*)' at the end of the title.

(*Page* 6, *Table* 1, *footnote marked* '||') — Substitute the following for the existing:

'||Methods of sampling and test for natural and synthetic perfumery materials : Part 5 Determination of refractive index (*second revision*).'

(*Page* 7, *clause* A-2.1, *line* 2) — Substitute 'IS 248 : 1987*' *for* 'IS : 248 - 1978*'.

(*Page* 7, *footnote marked* '*') — Substitute 'Specification for sodium bisulphite (sodium metabisulphite) (*fourth revision*)' for the existing.

(PCD 18)

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Indian Standard
SPECIFICATION FOR CITRAL
(First Revision)

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(*Continued on page* 11)

Indian Standard

SPECIFICATION FOR CITRAL

(First Revision)

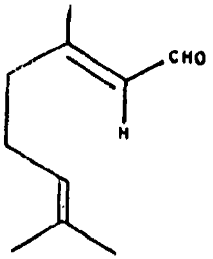
0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 26 September 1981, after the draft fiinalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Ptroleum, Coal and Related Products Division Council.

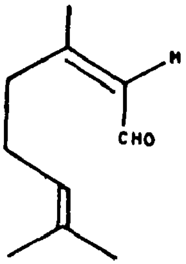
0.2 This standard was first published in 1961. The Sectional Committee responsible for the preparation of this standard felt that the standard should be revised in view of the trade practices in perfumery technology and to suit the material currently being manufactured and sold in the country.

0.3 In the present revision, the requirement for solubility of the material in sodium bisulphite and the corresponding method of test, which was deleted through Amendment No. 1 September 1972 from the original standard has been included again. Gas chromatographic method which is progressively coming into use in the country has been prescribed for the determination of citral. Furthermore, separate standard on 'Methods of sampling and test for natural and synthetic perfumery materials' have been prepared for each method of test and references have been made accordingly.

0.4 Citral ($C_{10}H_{16}O$), as it occurs in nature and also commercially available, is invariably a mixture of two geometric isomers, namely, geranial and neral. The structural formulae of the two forms of citral, namely, geranial and neral may be represented as follows:



GERANIAL
(α-CITRAL)



NERAL
(β-CITRAL)

(Molecular Mass 152.24)

0.5 Citral occurs to a considerable extent in various essential oils particularly as the main constituent of the oil of lemongrass and also of the oil of *Litsea Cubeba* which is produced mainly in China. To a small extent citral occurs also in various leaf and fruit oils of plants of genera *citrus*.

0.6 Citral in commerce is available mainly in two grades, namely, Grade 2 and Grade 1. Grade 2 is obtained by fractional distillation of lemongrass oil, and citral of this grade is contaminated with some other constituents of the parent oil which distil over along with citral. Grade 1 citral is produced by purifying Grade 2 citral through its bisulphite adduct This grade of citral is suitably stabilized to guard against any appreciable decrease in its purity during storage. Citral is also produced synthetically from β — pinene.

0.7 In the preparation of this standard assistance has been derived from the following publication:

> EOA 15 Standard for Citral Pure, published by Essential Oil Association of USA, New York.

0.8 The relevant Indian Standard on test methods (IS : 326-1968*) is under revision wherein each test method is being published as an independent part under the same IS designation, namely, IS : 326- (*second revision*). Till such time as all the test methods given in IS : 326-1968* are revised and published, for some test methods IS : 326-1968* would still be valid.

0.9 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for two grades of citral largely used in trade and industry. The material is used in flavours, perfumes and also as an intermediate for the manufacture of ionones and methyl ionones.

---

*Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

†Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions, given in IS : 6597-1972[*] shall apply.

3. GRADES

3.1 There shall be two grades of citral, namely:

Grade 1, and Grade 2.

4. REQUIREMENTS

4.1 Description

4.1.1 The material shall be a clear liquid; free from sediment, suspended impurities, separated water and adulterants; when examined as prescribed in IS : 326 (Part II)-1980[†].

4.2 Solubility — Both the grades of material shall be soluble in 7 volume of ethanol (70 percent by volume) when tested as prescribed under **8** of IS : 326-1968[‡].

4.3 The two grades of the material shall also comply with the requirements given in Table 1.

5. SAMPLING

5.1 Representative samples of the material, each sample containing not less than 50 ml, shall be drawn as prescribed under **3** of IS : 326-1968[‡].

6. TESTS

6.1 Tests shall be conducted as prescribed in IS : 326-1968[‡], IS : 2284-1963[§] and Appendices A and B. Reference to relevant parts and clauses is given in col 5 and 6 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977[||]) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

---

[*] Glossary of terms relating to natural and synthetic perfumery materials.

[†] Methods of sampling and test for natural and synthetic perfumery materials: Part II Preliminary examination of perfumery materials and samples (*second revision*).

[‡] Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

[§] Method for olfactory assessment of natural and synthetic perfumery materials.

[||] Specification for water for general laboratory use (*second revision*).

TABLE 1 REQUIREMENTS FOR CITRAL

(*Clause* 4.3)

SL No.	CHARACTERISTIC	REQUIREMENT		METHODS OF TEST, REF TO	
		Grade 1	Grade 2	Indian Standard	Appendix
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour and appearance	Colourless to pale yellow liquid	Pale yellow liquid	IS : 326 (Part II)-1980[*]	—
ii)	Odour	Citrus-Lemon like	Citrus-Lemon like	Cl 4 & 5 of IS: 2284-1963[†]	—
iii)	[‡]Relative density at 27°/27°C	0·885 0 to 0·887 5	0·882 0 to 0·885 9	IS : 326 (Part III)-1980[*]	—
iv)	[§]Refractive index at 27°C	1·483 0 to 1·486 0	1·480 0 to 1·486 0	Cl 7 of IS: 326-1968[‖]	—
v)	Aldehyde content expressed as sodium meta bisulphite solubility, at 27 C percent by volume, *Min*	100	90	—	A
vi)	Total carbonyl compounds calculated as citral, percent by mass (using 1·0 g sample), *Min*	97	88	—	B

[*] Methods of sampling and test for natural and synthetic perfumery materials:
Part II Preliminary examinations of perfumery materials and samples (*second revision*).
Part III Relative density (*second revision*).

[†] Method for olfactory assessment of natural and synthetic perfumery materials.

[‡] The correction factor for relative density for each degree Celsius change in temperature is 0·000 80.

[§] The correction factor for refractive index for each degree Celsius change in temperature is 0·000 45.

[‖] Methods of sampling and test for natural and synthetic perfumery materials (*first revision*).

7. PACKING AND MARKING

7.1 Packing — The material shall be supplied in air-tight containers, preferably amber coloured or opaque glass bottles, permitting a minimum air-space, as agreed to between the purchaser and the supplier.

7.2 The material shall be protected from light and stored in cool and dry place.

7.3 Marking — The material shall be marked with the following information:

a) Name and grade of the material;
b) Name of the manufacturer and recognized trade-mark, if any;
c) Net mass of the material; and
d) Batch number.

7.3.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

APPENDIX A

[*Table* 1 *Item* (v)]

DETERMINATION OF SOLUBILITY OF CITRAL IN SODIUM BISULPHITE SOLUTION

A-0. GENERAL

A-0.1 The reaction of sodium bisulphite with the carbonyl compounds yieldsa bisulphite addition compound, which is soluble in water.

A-1. APPARATUS

A-1.1 Cassia Flask — one, 150 ml.

A-1.2 Water-Bath

A-2. REAGENTS

A-2.1 Sodium Bisulphite Solution — A freshly prepared 30 percent solution (*m/m*) of sodium bisulphite, technical (*see* IS : 248-1978*).

---

*Specification for sodium bisulphite (sodium metabisulphite) (*third revision*).

A-3. PROCEDURE

A-3.1 Take 10 ml of the material in a 150-ml Cassia flask and add 10 ml of 30 percent sodium bisulphite solution. Shake the flask in a boiling water-bath for two minutes. The material usually solidifies at this stage. If it does not, continue shaking the flask, again immerse in a boiling water bath for two minutes. Add an additional 50 ml of sodium bisulphite solution and shake the flask in water bath until the material on being completely reacted is apparently clear. Add enough hot bisulphite solution to raise the meniscus within the graduated portion of the flask. Immerse the flask in the boiling water bath for 10 minutes, then remove. Cool the contents of the flask to 27°C.

A-3.1.1 The material of Grade 1 shall be taken as conforming to the requirement of the test if no oil separates or smears on the surface of the flask when it is tested as in **A-3.1**. On smelling the solution, no traces of any foreign odour are detected.

A-3.1.2 The material of Grade 2 shall be taken as conforming to the requirement of the test if not more than one millilitre of citral separates when it is tested as in **A-3.1**. Foreign odours may also be perceptible.

APPENDIX B

[*Table* 1, *Item* (vi)]

GAS CHROMATOGRAPHIC ANALYSIS FOR CITRAL

B-0. GENERAL

B-0.1 The chromatographic conditions given here are for guidance only. Variations in conditions to arrive at the typical chromatographic separation shown in Fig. 1 are possible.

B-0.2 Outline of the Method – A sample of the material is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

B-1. APPARATUS

B-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used.

A typical chromatogram using such a gas chromatograph is shown in Fig. 1 with following chromatographic conditions:

i) Column	Stationary phase : Carbowax 20 m, 10 percent by mass Solid Support : Chromosorb WAW 60-80 mesh	
ii) Carrier Gas	Hydrogen	
iii) Conditions	Column temperature Isothermal	: 170°C
iv) Injection Port	Temperature	: 200°C
v) Carrier Gas	Flow rate	: 40 ml/min
vi) Detector	Type : Thermal Conductivity; temperature	300°C
vii) Recorder	Span : 1 mV Chart Speed : 1 cm/min	
viii) Attenuation	4	

B-1.2 For Citral Grade 2, no representative chromatogram has been given as the material varies with requirements of the end user.

B-2. PROCEDURE

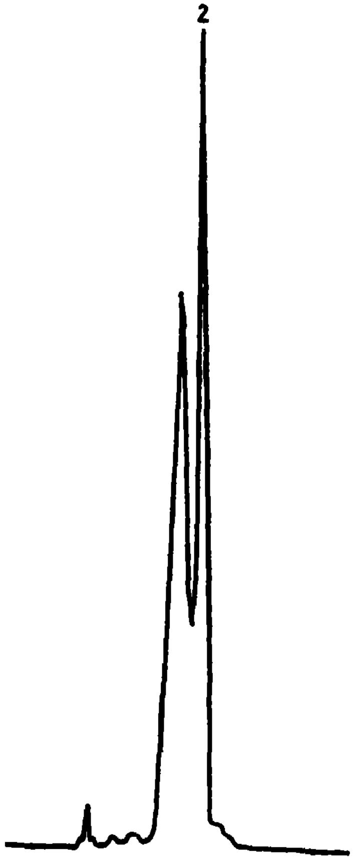
B-2.1 Conduct the flow of the carrier gas and inject sample at injection port where it is vaporized and well mixed with carrier gas. This is led into the chromatographic column wherein vaporized. The constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For the separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives the signal corresponding to the amount of particular constituent leaving the column. The detector signals, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

B-3. CALCULATION

B-3.1 Calculate the peak areas of individual constituents shown on chromatogram of the material, concentrations of the constituents may be determined on the basis of peak areas on chromatograms obtained with known amount of pure constituents using the same apparatus under identical conditions.

B-4. RESULTS

B-4.1 The typical chromatogram is shown in Fig. 1.



1. Citral beta (Neral)
2. Citral Alpha (Geranial)

FIG. 1 TYPICAL CHROMATOGRAM OF CITRAL GRADE 1

(*Continued from page* 2)

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Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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